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ELECTRICAL CONDUCTIVITY MEASUREMENTS OF HYDROXYLAMMONIUM NITRATE: DESIGN CONSIDERATIONS

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April 1986

US ARMY BALLISTIC RESEARCH LABORATORY
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The phenomenon of electrical conductance in aqueous salt solutions is of great interest to both experimental and theoretical scientists alike. Electrical conductivity can be used as a tool for elucidating the dynamics of ions in solutions. Our interest lies in the liquid structure and transport properties in aqueous solutions of hydroxylammonium nitrate (HAN), a major component in candidate liquid gun propellants. This report reviews the experimental considerations in making electrical conductivity (CONT'D)

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20. Abstract (Cont'd):

measurements on aqueous salt solutions, such as HAN. Conductance measurements as a function of HAN concentration are also reported; discussions as to the meaning of these curves and possible similarities with Raman spectra will be the subject of a future report.

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I. INTRODUCTION

For many nonmetallic substances such as aqueous salt solutions, the phenomenon of electrical conductivity occurs via the movement of "free ions." Materials of this kind are referred to as electrolytes. The conductivity of an electrolyte depends upon the material, concentration, temperature, and the geometry of the system with respect to the electrodes used in the measurement. It is desirable to remove the geometric dependence, which is done as follows. The conductance L is defined as the reciprocal of resistance and is expressed as ohms^{-1} (Ω^{-1}). The conductance of a homogeneous body of uniform cross section is proportional to the cross section A and inversely proportional to the length ℓ;

$$L = \frac{1}{R} = \frac{\bar{L}A}{\ell} \text{ where } \bar{L} = \frac{1}{R}\frac{\ell}{A} = \frac{k}{R} . \qquad (1)$$

$\bar{L}$ is the specific conductance and $k = \ell/A$ is the cell constant. Thus, the specific conductance of a solution in a cell of arbitrary design can be obtained by first determining the cell constant and then measuring the resistance of the solution in that cell. The cell constant can be determined by either a geometry measurement or by measuring the resistance of a solution of known specific conductance. Solutions of potassium chloride are generally used for this purpose. The specific conductance, because of its sensitivity to the concentration (c) of the conducting species, is a poor parameter to compare the intrinsic behavior of various electrolytes. This concentration dependence can be removed by use of molar conductance M which is defined as

$$M = \bar{L}/c$$

where c is in gram-moles per cm^3. If c is given in gram-moles per liter of solution then

$$M = \frac{1000\bar{L}}{c} , \qquad (2)$$

In addition, the ability of an electrolyte to carry current depends not only on the ionic concentration, but the ionic valence (n_e) as well. The equivalent conductance, Λ can now be defined as

$$\Lambda = M/n_e .$$

For the class of salt solutions under consideration here, the valency will be one, thus the molar and equivalent conductances are identical.

Now that a few of the basic parameters of conductance have been defined, we will discuss some of the experimental problems encountered during the course of the measurement.[1] The circuit used for measuring conductance is extremely simple and is shown in Figure 1. In this figure, A and V are Fluke Model 8060A RMS multimeters, OSC is a Tektronix FG 502 function generator, and C is the conductivity cell.

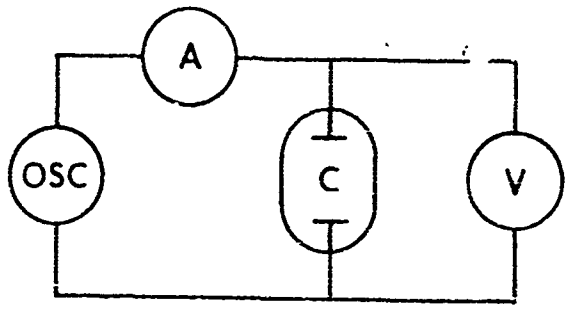

Figure. 1. Electrical Circuit Used for Performing Conductivity Measurements on Aqueous Salt Solutions.

The multimeters have sufficient sensitivity to measure voltages and currents to within 0.1% precision over the ranges of interest. The oscillator produces a sine wave and is continuously variable from 1 to 10^7 Hz. The ability to vary the frequency is very useful in determining the proper operations of the conductance cell, which will be discussed shortly.

II. CELL DESIGN FACTORS

Discussions in the literature have indicated that as many as seven different cells are required to accurately measure the conductance of electrolytic solutions over the entire concentration range from infinite dilution to saturation. For expedience it was decided to use one cell that would have a minimal error over most of the concentration range with emphasis on accuracy for the more concentrated solutions which are representative of actual liquid propellants.

Sasse'[2] suggested the use of a Hamilton syringe for the conductivity cell. The Hamilton syringe is attractive because the stainless steel needle and plunger form the electrodes and the distance (ℓ) can be varied by movement of the plunger. The cell size selected takes a maximum of 100 microliters of sample, and is easily immersible in a thermostatically controlled bath for temperature dependence studies. This type of cell was used in Figure 1.

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1. Many of these phenomena have been discussed in the 1928-1935 literature. A good review is given in the book Electrolyte Solutions by Robinson and Stokes, London Butterworths Scientific Publications, 1955.

2. R.A. Sasse', private communication, 1985.

The resistance values obtained as a function of frequency and distance were plotted for various concentrations of aqueous hydroxylammonium nitrate (HAN) solutions. The HAN solutions we obtained were nominally 13 M, and we accurately diluted them to produce the other concentrations. It should be mentioned that HAN is dissolvable in water to concentrations greater than 17 M at room temperature. Figures 2-4 display such data for 0.10, 1.0, and 13 M HAN solutions, respectively. As can be seen, the resistance is definitely a function of frequency; furthermore, the resistance does not scale linearly with cell length (ℓ). This non linearity is shown in Table 1 by ratioing the resistance values with the ℓ ratio 4.5 cm/1.5 cm for the three concentrations at a fixed frequency (10^3 sec^{-1}). The resistance values for the 1.0 and 13 M HAN solutions are observed to be similar, and it is also observed that the dependence on frequency and length is correspondingly similar. At 0.10 M, the dependence on frequency is less and the cell resistance ratio is closer to the linear value of 3.00.

TABLE 1. Variation of Resistance Ratios as a Function of Concentration for 100 Microliter Hamilton Syringe Conductivity Cell at 1 KHz

Cell Length Ratio	Cell Resistance Ratio		
	0.10 M	1.0 M	13 M
$\frac{4.5}{1.5} = 3.00$	$\frac{56.92}{21.13} = 2.69$*	$\frac{4.432}{2.235} = 1.98$*	$\frac{3.230}{1.805} = 1.79$*
	2.88**	3.06**	3.03**

* - These values are obtained at f=1000 Hz.

** - These values are obtained by extrapolating Figures 2-4 to infinite frequency, which results in an intercept on the ordinate.

Clearly, this is an unsuitable cell design to use in making reliable conductivity measurements on HAN solutions. In order to construct a more appropriate cell, the physical reasons for these variations need to be understood.

It has been known for many years that polarization effects can occur between electrolytic solutions and the electrodes. In fact, conductivity measurements are made with alternating current to minimize this problem. Through detailed experimental studies and models of diffusion at the electrode it was found[3] that the polarization effect causes a resistance in series with the electrolytic solution and furthermore that this resistance varied inversely with the square root of the frequency (f). Taking account of the polarization effect, one can write

3. G. Jones and S. Christian, "The Measurement of the Conductance of Electrolytes. VI. Galvanic Polarization by Alternating Current," J. Am. Chem. Soc., Vol. 57, p. 272, 1930.

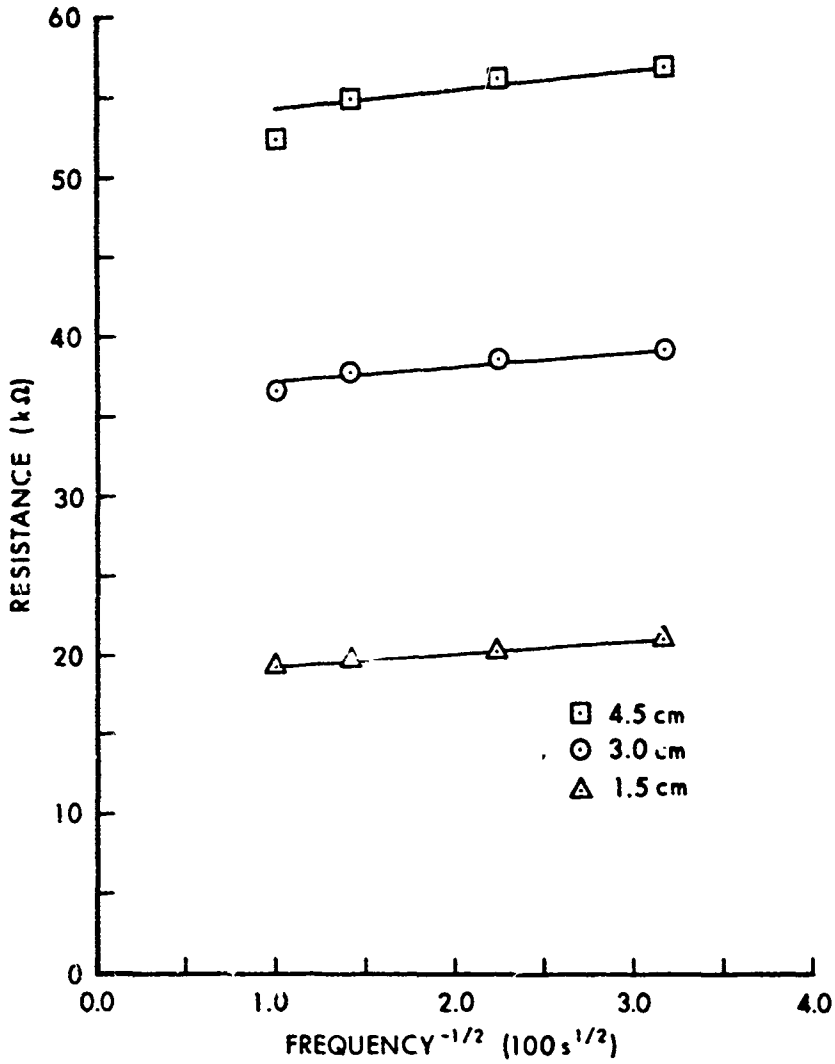

Figure 2. Resistance Versus Frequency$^{-1/2}$ for a 0.10 M HAN Solution Contained in a 100 Microliter Hamilton Syringe. Three Path Lengths (ℓ) are Displayed. T = 20°C.

$$R_t = R_s + R_p/\sqrt{f} \qquad (3)$$

for the total resistance (R_t) of a conductivity cell, where R_s is the solution resistance and R_p is the resistance created by polarization effects. As f approaches infinity R_t approaches R_s, however f cannot be increased arbitrarily high because of another effect to be discussed shortly. Figures 2-4 all show that R_t indeed decreases with increasing frequency indicating that the Hamilton syringe cell is influenced by polarization effects. The cell length and cell resistance ratios for the Hamilton syringe can also be explained in this context. The most dilute solution (0.10 M HAN) gives a cell resistance ratio closest to the cell length ratio (see Table 1). The reason for this result is that the solution resistance, R_s, is highest in this case providing the major contribution to R_t. If one does a straight line extrapolation to infinite frequency, the cell resistance ratios obtained from the intercepts provide cell resistance ratios which are in line with the cell

length ratio (again see Table 1). Although measurements of R_s could be made from such extrapolations, it would be much better to design a cell that minimizes the polarization effects.

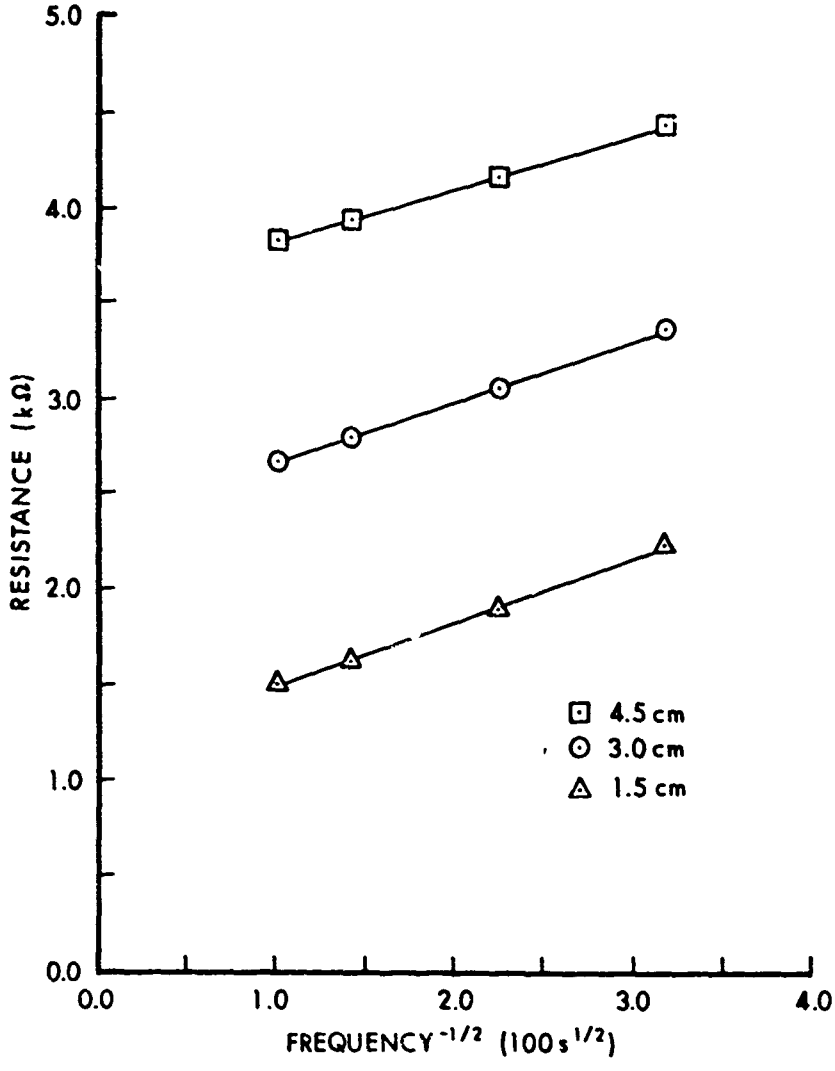

Figure 3. Same as Figure 2, Except that a 1.0 M HAN Solution is Used.

One can see from equation (3) that increasing R_s makes R_p a smaller contribution. This can be accomplished by increasing the value of the cell constant ($k = \ell/A$). However, increasing the cell constant by decreasing A has been shown to create adverse effects. R_p itself is minimized by use of large area inert electrodes usually made from platinum. In many cases these electrodes are electroplated with platinum black to increase the effective surface area and this has been shown to further decrease R_p. Thus ℓ is the choice variable to use in increasing the cell constant.

There is a danger in going to extremely large values of R_s because of shunting effects. That is, impedance paths may appear which become the same order of magnitude as the resistive path through the solution. An equivalent circuit for the conductivity cell can be represented by Figure 5,

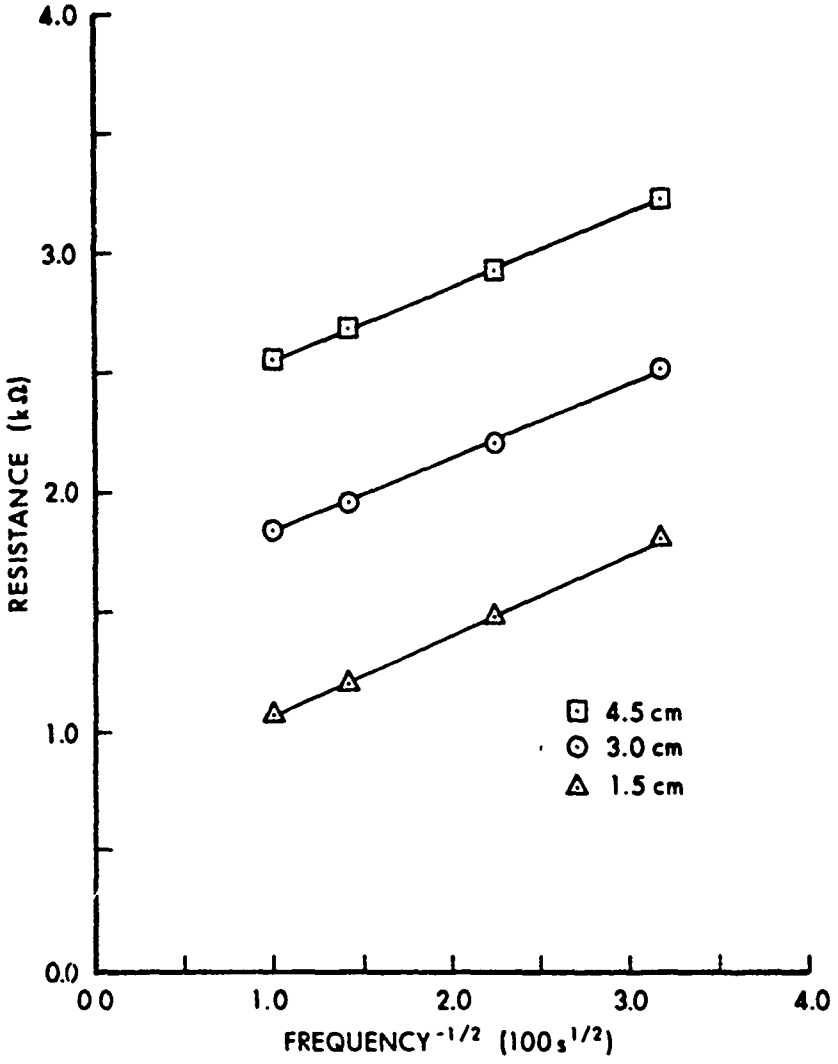

Figure 4. Same as Figure 2, Except that a 13 M HAN Solution is Used.

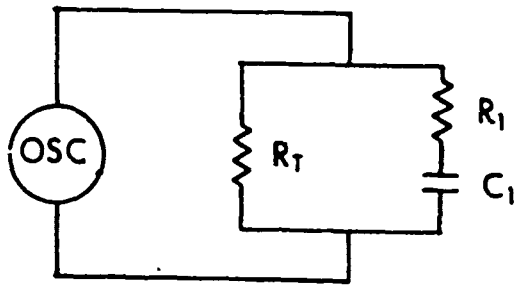

Figure 5. An Equivalent Circuit Representing the Shunting Effect on a Conductivity Cell

where R_1 and C_1 are shunting resistances and capacitances. The circuit impedance (Z) is given by

$$\frac{1}{Z} = \frac{1}{R_t} + \frac{1}{R_1 + X_C}$$

where $X_c = \frac{1}{2\pi f C_1}$ is the capacitive reactance.

Separating out the real part of the impedance (R) we have after some algebra

$$R = R_t \left(1 - \frac{R_t R_1 + R_t^2}{(R_t + R_1)^2 + X_C^2}\right) .$$

Since all of the values contributing to the second term on the right are positive, the shunt always lowers the resistance, an effect opposite to that of polarization. To simplify matters a bit, assume $R_1=0$, then

$$R = \frac{R_t}{1+4\pi^2 f^2 C^2 R_t^2}$$

we see the capacitance effects are minimized by keeping the term $4\pi f^2 C^2 R_t^2 \ll 1$. Practically speaking, one does not want to use extremely large values of f or R_t. Details of the shunting effect are discussed by Jones and Bollinger.[4]

In summary, it is found that for electrolytic solutions of low resistance, i.e., concentrated solutions, one has to watch for polarization effects, while for electrolytic solutions of high resistance, i.e., dilute solutions, shunting effects can become important. It would be desirable to have a conductivity cell that exhibited a frequency independent resistance over a substantial frequency range, indicating the above-mentioned effects are negligible. The next section describes such a cell together with some conductance measurements.

III. TEST AND MEASUREMENT

From the arguments contained in the last section, a new conductivity cell was constructed from a glass U-tube having a path length of 58 cm, an inside diameter of 0.70 cm, and an outside diameter of 0.90 cm. The electrodes are 0.6 cm diameter platinum discs which are held in position by a non-conductive support which rests on the top of the U-tube. This arrangement allows for easy removal of the electrodes for cleaning purposes.

---

4. G. Jones and G. Bollinger, "The Measurement of the Conductance of Electrolytes. III. The Design of Cells," J. Am. Chem. Soc., Vol. 53, p. 411, 1931.

There are several dramatic geometric changes in the cell design over the 100 microliter Hamilton syringe; the path length ℓ is about 10 times larger and the electrode area is about 15 times larger. The cell constant for this cell is calculated from the geometry to be 150.7 cm^{-1}. The conventional way to obtain the cell constant is to measure the resistance of a known solution. Using a .100 N KCl solution, the cell constant is measured to be 145.3 cm^{-1}. This agreement is quite good considering the approximate nature of the geometric measurement. A logical next step in testing this cell is to look at the frequency dependence of the resistance for various solutions. Figure 6 illustrates three cases: a 0.10 M, 1.0 M, and 13 M HAN solutions. We can see from the figure that the resistance is found to be independent of frequency over the range 500 to 10,000 Hz. This is indeed an improvement over the Hamilton syringe. As a final test, several concentrations of aqueous sodium nitrate solutions were prepared and their resistances were measured with the cell. These experimental results together with published data for the conductance of aqueous $NaNO_3$ are given in Figure 7. Excellent agreement is obtained between our measurements and published results. These experimental checks indicated that this cell was functioning properly; we therefore used this cell to obtain conductance measurements on HAN as a function of concentration.

Figures 7 and 8 illustrate this data. Figure 7 is a plot of specific conductivity of HAN versus concentration. Data for aqueous $NaNO_3$ is shown as well to indicate the similarity of this conductivity data with that of HAN. The solubility of $NaNO_3$ in H_2O is much less than that for HAN; consequently, data for aqueous $NaNO_3$ only goes to approximately 6 M. Figure 8 shows the equivalent conductance as a function of the square root of the concentrations. There are several reasons for plotting data in this fashion. The equivalent conductance shows what is happening on a "per ion" basis, and when plotted versus $c^{1/2}$, can be compared with the dilute solution theories of Debye and Hückel and Onsager. These theories say in effect that the electric forces between ions tend to maintain a space and oppose the motion of an ion under the influence of an outside electric field, and thus cause a decrease in the conductance proportional to the $c^{1/2}$. Further measurements of conductance in dilute HAN solutions will be performed. The meaning of the conductance curves and possible similarities with conclusions drawn from Raman spectra are discussed in other reports.[5,6]

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5. S.W. Bunte, J.A. Vanderhoff, and P.M. Donmoyer, "Electrical Conductivity Measurements on Hydroxylammonium Nitrate, LGP 1845 and LGP 1846," Proceedings of the 22nd JANNAF Combustion Meeting, to be published.

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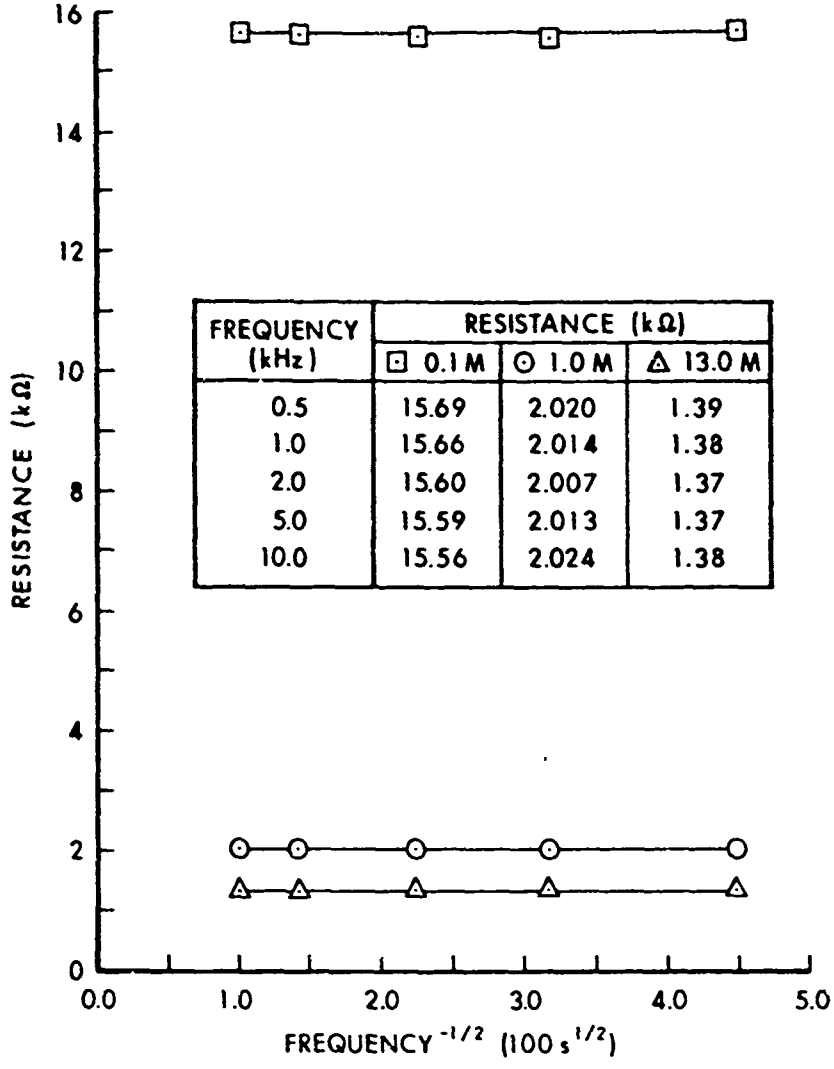

FREQUENCY (kHz)	RESISTANCE (kΩ)		
	⊡ 0.1 M	⊙ 1.0 M	△ 13.0 M
0.5	15.69	2.020	1.39
1.0	15.66	2.014	1.38
2.0	15.60	2.007	1.37
5.0	15.59	2.013	1.37
10.0	15.56	2.024	1.38

Figure 6. Resistance Versus Frequency$^{-1/2}$ for 0.10, 1.0, and 13 M HAN Solutions Using the Constructed U-Tube Conductivity Cell. T = 20°C.

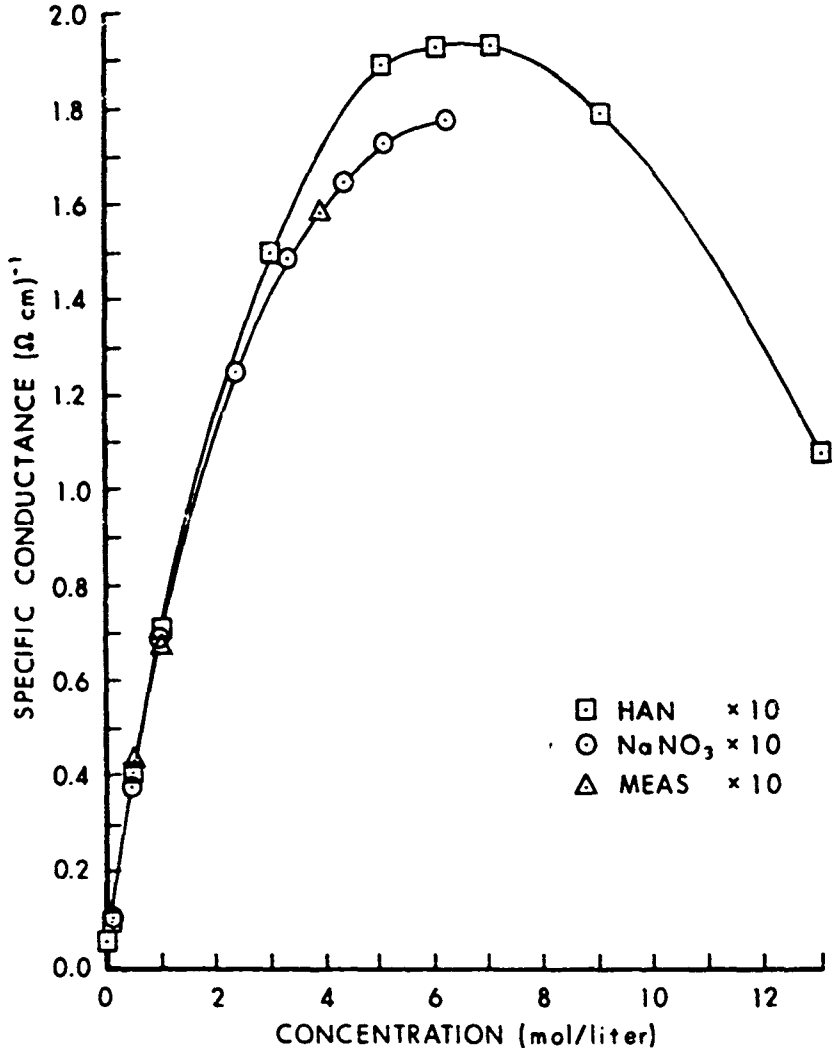

Figure 7. Specific Conductivity Versus Concentration for Aqueous HAN and $NaNO_3$. The $NaNO_3$ Published Data (0) is From the CRC Tables. The U-Tube Conductivity Cell is Used and T = 20°C.

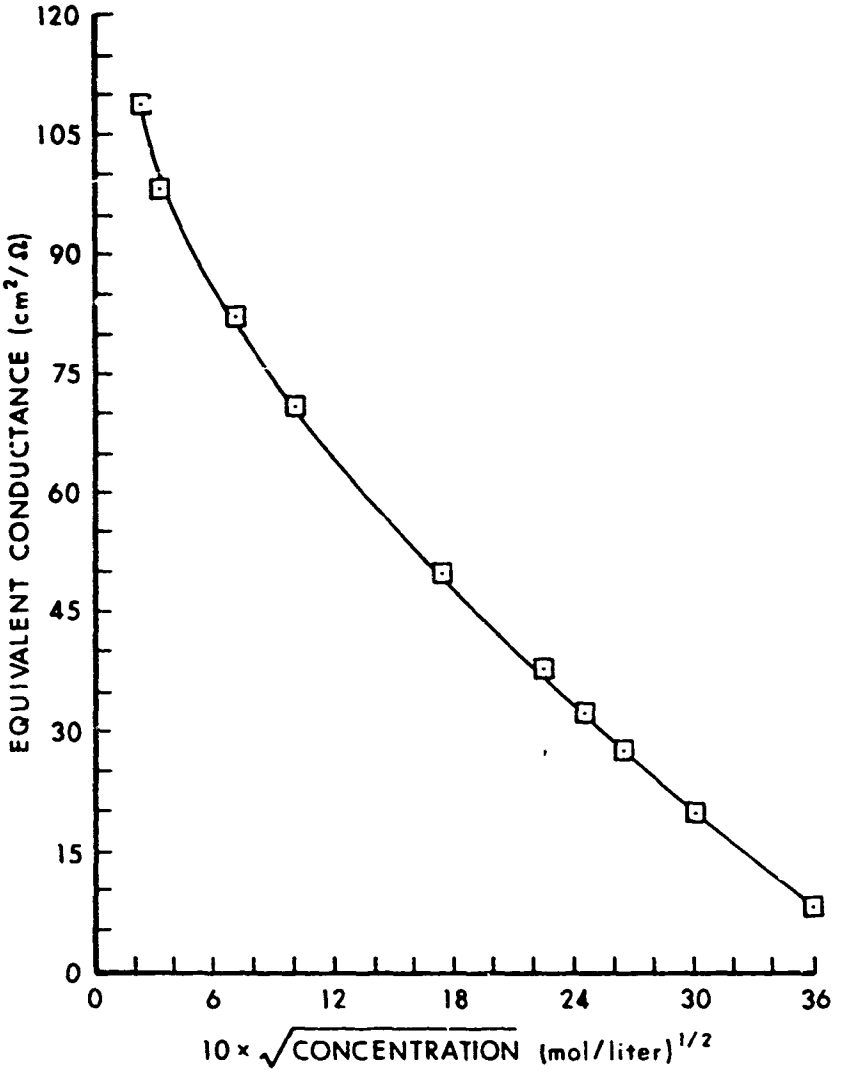

Figure 8. Equivalent Conductance Versus (Concentration)$^{1/2}$ for Aqueous HAN Using U-Tube Conductivity Cell. T = 20°C.

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2. R.A. Sasse', private communication, 1985.

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